

THE DETERMINATION OF ORGANIC MATTER IN SOILS BY MEANS OF HYDROGEN PEROXIDE¹

By W. O. ROBINSON

*Associate Chemist, Division of Soil Chemical Investigations, Bureau of Soils,
United States Department of Agriculture*

INTRODUCTION

The organic matter of soils is generally determined by converting it to carbon dioxide by means of moist and dry combustion. The carbon dioxide thus evolved is then calculated to organic matter by the use of a conventional factor, 0.471. It has been found, however, that the percentage of carbon in the organic matter of soils varies widely. Some doubt, therefore, is cast on the accuracy of the combustion methods, as applied to any particular soil. For this reason a new method which does not involve the use of an empirical factor is desirable.

Inasmuch as several investigators have reported that a large part of the soil organic matter is decomposed by hydrogen peroxide, it seemed possible that the organic matter might be determined by treating a sample of soil with hydrogen peroxide and noting the loss in weight. A treatment with hydrogen peroxide, unlike treatment by the combustion methods, would not be expected to affect the combined water content or appreciably change the weight of the inorganic material.

In testing the use of hydrogen peroxide for determining organic matter, information has been obtained regarding the decomposability of various kinds of soil organic matter; and also, concerning the accuracy of the conventional factor, 0.471, when applied to the organic matter of different soils.

REVIEW OF PREVIOUS WORK

Coppenrath, Hasenbäumer, and König (2)² and also May and Gile (6) have shown that the quantity of oxygen set free from the hydrogen peroxide used is roughly proportional to the quantity of organic matter in the soil.

König, Hasenbäumer, and Grossmann (5) found that hydrogen peroxide converted from 60 to 90 per cent of the total carbon of the soil to carbon dioxide.

Peterson (7), when studying the organic phosphorus of soils, was the first to determine the loss in weight of the soil material caused by the destruction of the organic matter by hydrogen peroxide. His method consisted in digesting the soil with dilute hydrogen peroxide in a covered beaker until the weight became constant. On comparing the loss in weight with the quantity of organic matter determined by combustion, he concluded that hydrogen peroxide

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² Reference is made by number (italic) to "Literature cited," p. 355.

destroyed about 90 per cent of the soil organic matter. Peterson did not determine whether any carbon was left in the digested residues; thus it is not known whether failure of the two methods to agree was due to incomplete oxidation or to organic matter which has a different percentage of carbon than that assumed when the conventional factor is used.

Robinson (9) proposed the use of hydrogen peroxide in the mechanical analysis of soils to destroy the organic matter which prevented the deflocculation of the soil particles. He observed that the destruction of the organic matter was not complete. Later he and Jones (10) found that cotton fiber, crude fiber from straw, etc., were not decomposed by hydrogen peroxide under their experimental conditions, whereas humus, or organic matter which had been extracted from soils by ammonia was completely decomposed. They therefore proposed the use of hydrogen peroxide to differentiate between the humified and nonhumified organic matter of the soil. The humified organic matter was determined by subtracting the ignition loss of the soil after treatment with hydrogen peroxide from the ignition loss of the untreated soil. They found that the humified organic matter varied from 3.4 to 57.5 per cent in the mineral and peat soils of Wales, and that it constituted from 42.3 to 82.3 per cent of the total organic matter, as determined by the loss on ignition. The percentage of organic matter destroyed is doubtless somewhat too low, since the loss on ignition is almost invariably greater than the organic matter contained in a soil. Robinson and Jones took precautions to minimize this error by selecting soils free from carbonates and, presumably, low in combined water, since they were of low clay content.

THE HYDROGEN PEROXIDE METHOD

In the hydrogen peroxide method the soil organic matter is determined from the loss in weight caused by digesting the soil with hydrogen peroxide. The method adopted in this investigation is described as follows:

About 1 gm. of ordinary soil or 0.2 gm. of peat was accurately weighed into a tall 250 c. c. beaker, to which 10 c. c. of water and 10 c. c. of 30 per cent hydrogen peroxide³ were added. The beaker was covered and watched for a few minutes. If the reaction was not too vigorous,⁴ the beaker was placed on a steam bath and the contents digested until no more bubbles evolved. If the reaction is too vigorous the method can not be applied without modification to the particular soil. The digestion was usually complete within 1 hour, the peats and mucks requiring more time. After the bubbles ceased no more hydrogen peroxide remained, as is shown by the permanganate test. The beaker was frequently manipulated during the digestion in order to bring the lighter particles of organic matter in contact with the solution as much as possible. After it had settled the solution was filtered through a thick and compact asbestos pad in a special Gooch crucible. Filtration is necessary to separate

³ This hydrogen peroxide contained from 30 to 33 per cent of H_2O_2 . The ash content of 0.001 per cent was disregarded. A small quantity of preservative present contained nitrogen and carbon. Suitable allowances were made for the nitrogen and carbon in some of the details of this study.

⁴ A violent reaction with the evolution of heat shows the presence of manganese dioxide (MnO_2) or chromium sesquioxide (Cr_2O_3) in the soil. If the quantity of manganese dioxide is small (say less than 0.25 per cent), it can be destroyed by evaporating with a very small excess of oxalic acid, and the soil may then be treated as indicated above. If much manganese dioxide is present, this method is not applicable.

from the residue some soluble, undecomposed organic matter and ammonium salts which are always present in solution. The residue was washed a little, and the filtrate and washings were evaporated in a platinum dish, ignited and weighed. This was necessary since the weight of the ash of the soluble matter must be added to the weight of the residue.

The filtrate should be practically clear. Occasionally heavy clay soils will give a turbid or muddy filtrate. If passing the suspension through the pad a second time without allowing the pad to become dry fails to remove the suspended matter, certain corrections must be made. The weight of the ignited suspended matter can not be directly added to the weight of the residue in the Gooch crucible, since the clay loses about 10 per cent of combined water on ignition. Separate estimates of clay and soluble inorganic matter must be made, so that the ignited clay can be calculated to its weight at 110° C. This is done as follows: The filtrate is made up to a definite volume and is allowed to settle. The clear, upper half is evaporated, ignited, and weighed, giving one-half of the weight of the soluble inorganic matter. The turbid, lower half is treated in the same manner. The difference between these two weights gives the weight of ignited clay. This weight is increased by one-ninth to give the weight of the clay dried at 110° C. The corrected weight of the clay and the determined weight of the soluble, inorganic material are then added to that of the filtered residue.

The insoluble matter remaining on the pad was dried for 18 hours at 110° C. and weighed. The moisture determination in the untreated soil material was made at the same time. The difference in weight between the moisture-free material and the total weight of the dried residue plus the ash obtained from the solution was calculated as organic matter.

The concentrations and quantities of peroxide most favorable for the complete decomposition of the organic matter were selected after several trials. The results of these trials on four soils, high and low in organic matter, are given in Table 1.

TABLE 1.—*Effect of hydrogen peroxide in different concentrations on the decomposition of organic matter in different types of soil*

Types of soil	Depth at which soil sample was taken	Decomposition solution used		Organic matter decomposed
		30 per cent H ₂ O ₂	Water	
	Inches	C. c.	C. c.	Per cent
Velasquez loam	12 to 32	10	-----	12.64
Do.	do.	10	10	12.94
Do.	do.	10	50	12.73
Do.	do.	20	20	13.00
Do.	do.	40	40	13.12
Ontario loam	0 to 12	10	-----	3.01
Do.	do.	10	10	2.82
Do.	do.	10	50	3.19
Carrington loam	do.	10	10	3.93
Do.	do.	20	20	3.63
Do.	do.	40	40	3.58
Peat	46 to 70	10	-----	87.1
Do.	do.	10	50	90.7
Do.	do.	20	20	86.6
Do.	do.	40	40	89.9

It is evident that accurate determinations of the dried weights of the soil and digested residues are very important. Considerable difficulty was experienced in drying the residue to constant weight in an ordinary Gooch crucible on account of the very hygroscopic nature of some of the materials. By providing the crucibles with tightly fitting ground-on covers, satisfactory results were obtained. Pyrex-glass crucibles were used.

The quantities of organic matter destroyed by hydrogen peroxide show no consistent variation with increasing quantities or concentrations of the reagent. The differences are about the same as are obtained between duplicates, although the duplicates agreed more closely in subsequent work, as the technique improved with practice. Evidently the quantity of hydrogen peroxide specified in the procedure was sufficient to produce maximum decomposition of the organic matter.

Although tests showed that nothing was to be gained by the use of different concentrations of hydrogen peroxide, it was not known whether more organic matter would be decomposed by additional treatments with the reagent. Two different soils were therefore subjected to successive treatments with 20 c. c. of the 15 per cent hydrogen peroxide used in the procedure. The results are shown in Table 2.

TABLE 2.—*Effect of successive treatments by hydrogen peroxide on quantity of organic matter decomposed in soils of different types*

Type of soil	Number of treatments with H ₂ O ₂	Organic matter decomposed
		<i>Per cent</i>
Velasquez loam.....	1	12.64
Do.....	2	12.81
Do.....	3	12.65
Peat.....	1	87.1
Do.....	2	87.8
Do.....	3	90.7

The data in Table 2 show no consistent increase in the quantities of organic matter decomposed by additional treatments with hydrogen peroxide. Later work showed that, under an excessively prolonged treatment, slightly more organic, or carbonaceous matter could be decomposed by hydrogen peroxide, but the process was too tedious to be adopted as a practical method. It is thought that the procedure adopted gives as complete decomposition of the organic matter as is practicable.

COMPOSITION OF SOIL MATERIAL MADE SOLUBLE BY HYDROGEN PEROXIDE

In describing the hydrogen peroxide method it was stated that the filtrate from the peroxide digestion contained soluble matter for which corrections were made.

About half of the matter made soluble was volatile at a dull-red heat. Part of the volatile matter was obviously organic, but its nature was not determined. König, Hasenbäumer, and Grossmann (5) found that formic and acetic acids were formed under similar

conditions. Ammonium salts also formed part of the volatile matter; in fact analyses of several of the salt solutions showed that practically all the soil nitrogen was converted into ammonium salts.

It is clear that the soluble, volatile matter should constitute a part of the organic matter indicated by the loss in weight of the material when treated with hydrogen peroxide. Thus, the weight of the residue from the peroxide digestion is not corrected for the soluble volatile matter. The ash remaining after the volatile matter is driven off, however, should be added to the weight of the residue. This correction for the ash, always made, ranged from 0.11 to 2.04 per cent, and was important in most cases. The ash does not seem to result from the solution of the soil inorganic matter, since the quantity of ash is not proportional to the quantity of hydrogen peroxide used, to the volume of the solution, or to the time of digestion. Evidently the ash comes from the organic matter which is decomposed, since different soils usually give quantities of ash proportional to the quantities of organic matter present. Furthermore, the ash yielded by different quantities of a particular soil is proportional to the quantity used.

The partial composition of the soluble inorganic matter present in the solution resulting from the digestion of three different soil materials with hydrogen peroxide is given in Table 3.

TABLE 3.—*Partial composition of inorganic matter in soils of different types made soluble by hydrogen peroxide (expressed as a percentage of the weight of soil)*

Type of soil	SiO ₂	Al ₂ O ₃ + Fe ₂ O ₃	CaO	MgO	MnO
	<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>	<i>Per cent</i>
Carrington loam.....	0.112	0.048	0.064	0.021	0.002
Hagerstown loam.....	.038	.022	.388	.044	.040
Velasquez loam.....	.073	.015	.168	.013	N. D.

Although the soluble inorganic matter of soils contains sulphates and chlorides also, the quantities of these elements were not determined. Evidently the constituents of the ash vary considerably in different soils. Lime is obviously one of the chief constituents. König, Hasenbäumer, and Grossmann (5) also found lime to be the chief soluble constituent under similar conditions. In some soils manganese may be the most important constituent of the ash. This was found to be so in the case of the Blakely and Amito loams.

It seems likely that most of the soluble inorganic matter has its origin in the destruction of the organic matter of the soil. In a sense it might be considered a part of the organic matter. However, it is left in the soil when the organic matter decays, and in all combustion methods it is neglected. It seemed best, therefore, to deduct the weight of the soluble, inorganic matter from the entire loss in weight caused by the digestion of the organic matter by hydrogen peroxide.

EXTENT TO WHICH CERTAIN KINDS OF ORGANIC MATTER ARE DECOMPOSED BY HYDROGEN PEROXIDE

Before determining the decomposability of the total soil organic matter it seemed best to study the action of hydrogen peroxide on some simpler organic materials, especially in view of the fact that

Robinson and Jones (10) reported that unhumified organic matter, such as cotton fiber and crude fiber, were not attacked by peroxide.

Preliminary tests made in the laboratory showed that the conclusion of Robinson and Jones regarding the nondecomposability of unhumified organic matter is true only under certain conditions. It was found that 15 per cent hydrogen peroxide had practically no effect on cotton fiber and crude fiber, provided they were digested in the absence of other material. However, when the digestion was carried out in the presence of soil both the cotton fiber and crude fiber were destroyed almost quantitatively.

Inasmuch as other investigators have found that all the soil organic matter is not decomposed by hydrogen peroxide, such material as charcoal, coal, and graphite were included in the substances tested, since they are likely to occur in soils and presumably are difficult to oxidize. Since the presence of soil material has such a marked effect on the decomposability of cotton fiber and crude fiber, the tests of the decomposability of various kinds of carbonaceous substances were made on samples mixed with a portion of the Orangeburg fine sandy loam subsoil. The proportion of organic matter decomposed was determined from the loss in weight of the sample in some cases, and in other cases, by measuring the carbon content before and after treatment. The results are given in Table 4.

TABLE 4.—*Decomposition of various kinds of carbonaceous matter by 15 per cent hydrogen peroxide in the presence of soil*

Kind of carbonaceous matter	Quantity decomposed
	<i>Per cent</i>
Coal, anthracite (100-mesh screen).....	10.2
Coal, bituminous (100-mesh screen).....	7.8
Coal, cannel (100-mesh screen).....	14.2
Charcoal in contact with soil but a short time ^a (100-mesh screen).....	29.8
Charcoallike particles separated from soil ^b (100-mesh screen).....	89.4
Cotton fiber.....	99.6
Crude fiber from wheat straw.....	98.4
Graphite, natural (100-mesh screen).....	0.0
Graphite schist separated from soil.....	0.0
Humus, extracted by ammonia.....	100.0
Lignite (100-mesh screen).....	98.7
Lubricating oil.....	66.4
Peat, mosslike.....	99.5
Peat, dark brown.....	97.0

^a Charcoal particles were separated from a cultivated soil where a large brush pile of hardwood had been burned about nine months before the charcoal was gathered.

^b Composite of several very small samples of charcoallike particles, separated from a number of soils and subsoils by Schreiner and Brown (11).

Table 4 shows that coals and graphite, in particular, were hardly attacked, and that charcoals were decomposed in varying degrees. More than half of a lubricating oil was destroyed, and the other substances were decomposed almost quantitatively.

It is certain that in the method adopted hydrogen peroxide can not be used to distinguish humified and nonhumified organic matter. Brown peat, mosslike peat, cotton fiber and crude fiber were almost completely decomposed under the conditions of this study. It is true that Robinson and Jones used weaker hydrogen peroxide and allowed a shorter time for digestion; but it is believed that the discrepancy between their results and those given in Table 4 is due

chiefly to the fact that they used hydrogen peroxide alone, whereas in the experiments described in this paper soil material, acting as a catalyzer, was always present during the digestion.

From these data it would be expected that the procedure adopted would result in the decomposition of all except the most resistant carbonaceous matter found in soils. The results for charcoal tend to show that different charcoals vary considerably in decomposability.

RESULTS OF ANALYSES OF DIFFERENT SOILS

A satisfactory procedure having been established, samples of material from a number of different soil types were analyzed for organic matter by the peroxide method.

Samples were selected from several soil types which are very high and several which are very low in organic matter. Three soil colloids and several very clayey subsoils were also selected with a view to studying the possible effects of colloid surfaces and of a high percentage of combined water on the accuracy of the method. To test the possible effect of the action of hydrogen peroxide on soil mineral matter, materials from several very deep subsoils low in organic matter were included. It was thought that if any oxidation of the mineral matter took place it would be greatest in the deep subsoil materials where natural oxidation is not so pronounced as in surface layers. Duplicate determinations were made on all samples. After the material had been treated with hydrogen peroxide, the residues were analyzed for undecomposed organic matter by the combustion method,⁵ and also for carbonates.⁶ These results are given in Table 5.

The results for organic matter, in Table 5, have not been corrected for the carbonates found in the residues. In each case the combustible matter found in the residue has been calculated to carbon, since it seemed likely that it contained a high percentage of carbon. All quantities are expressed as percentages of the weight of the original material used, dried at 110° C.

It is apparent, from columns 3 and 4 in Table 5, that the duplicate results obtained by the peroxide method agree about as well as those of other constituents, obtained by ordinary methods of soil analysis. To get closely agreeing duplicates it is necessary to observe extreme care in getting the dried weights. The sample should be weighed from a weighing bottle, and the Gooch crucible should be covered promptly just before transferring it from the drying oven to the desiccator.

The carbon dioxide of the carbonates in the residue evidently results from the action of hydrogen peroxide on the organic matter. The carbon dioxide, shown in the last column of Table 5, should properly be added to the organic matter found, since the residue is too heavy by just that quantity. However, it is apparent that such a correction would not be of any importance ordinarily. In 15 of the 26 materials analyzed this correction would be less than 0.15 per

⁵ An electric combustion furnace giving a maximum temperature of 1,100° C. was used. With a quartz combustion tube it was possible to heat the sample nearly to the maximum temperature, thus insuring complete combustion of graphite and other difficultly combustible carbon of low combustibility. The mixed gases from the furnace were dried by phosphorus pentoxide both before entering and in the carbon dioxide absorption apparatus.

⁶ Only one soil contained carbonates before treatment. A small quantity of carbonates is formed by the action of the hydrogen peroxide on the soil.

cent of the dry weight of the sample analyzed. The values given for organic matter in columns 3 and 4 of Table 5 have not been corrected for carbonates.

TABLE 5.—Organic matter in soils of various types as determined by the hydrogen peroxide method and the undecomposed carbon and the carbonates in the residues

Type of soil material	Depth at which soil sample was taken	Quantity of organic matter as determined by decomposition with H_2O_2 (duplicate results)		Quantity of combustible matter calculated as carbon, in residue from H_2O_2 treatment	Carbonates in the residues from the H_2O_2 treatment (calculated as CO_2)
		Per cent	Per cent	Per cent	Per cent
Amito loam, Mississippi	0-12	2.35	2.39	0.48	0.06
Do.	12-36	1.63	1.73	.37	.02
Blakeley loam, Georgia	0-12	2.14	2.24	.23	.24
Carrington loam, Iowa	0-12	¹ 3.59	¹ 3.59	.30	.00
Carrington loam colloid, Iowa	0-12	10.17	10.08	.41	.06
Cukre clay, Nicaragua, Central America	0-5	8.89	8.89	.67	.16
Do.	5-60	2.12	2.04	.28	.17
Hagerstown loam, Pennsylvania	0-8	4.78	4.67	1.90	.19
Houston black clay, Texas	0-12	3.39	3.49	.60	.20
Mosslike peat, Florida	0-8	95.60	95.50	.25	.02
Muck, Indiana	0-6	22.45	22.36	1.29	.75
Do.	6-12	12.17	12.38	.60	.33
Ontario loam, New York	0-12	¹ 3.00	¹ 3.00	.06	.05
Orangeburg fine sandy loam, Mississippi	10-36	.30	.41	.06	.00
Orangeburg fine sandy loam colloid, Mississippi	0-10	3.62	3.58	.38	.03
Lenox stony loam, Massachusetts	0-6	4.83	4.83	.50	.04
Peat, Florida	46-70	90.70	89.90	1.45	.37
Palmar clay, Colombia, South America	0-14	6.67	6.76	.26	.15
Do.	14-60	1.06	.95	.07	.06
Do.	60-72	.84	.74	.10	.06
Do.	72-96	.60	.61	.06	.02
Do.	96-120	.51	.51	.04	.00
Ranger loam, Georgia	0-10	2.00	1.80	.33	.00
Sharkey clay, Mississippi	0-4	3.98	3.72	.44	.21
Velasquez loam, Guatemala, Central America	12-32	² 12.90	² 12.90	.73	.27
Wabash silt loam colloid, Nebraska	0-15	4.60	4.59	.52	.09

¹ Average of 6 determinations.

² Average of 12 determinations.

The carbonaceous matter not oxidized bears no constant relation to the total quantity of organic matter present. In a few cases it is inconsiderable when calculated as carbon, amounting to less than 5 per cent of the total organic matter. In most of the materials, however, the undecomposed carbonaceous matter is considerable. It is evident, therefore, that the hydrogen peroxide method does not give an accurate determination of the total organic matter, that is, of the total quantity of carbon, or carbon compounds exclusive of carbonates.

NATURE OF THE UNDECOMPOSED ORGANIC MATTER

Since considerable quantities of organic matter or some substances containing carbon are not decomposed by the hydrogen peroxide treatment, the identification of these carbon-containing substances in the residues is important. If the residue after digestion contains only a definite kind of organic matter, the hydrogen peroxide method could be used to differentiate the two kinds of organic matter present in soils, and also to determine the quantity of the decomposable portion.

The quantity of organic matter left undecomposed is not dependent primarily on the state of subdivision, since the organic residue from the mosslike peat is less than the organic residues from the three soil colloids.

General knowledge concerning the soils treated was of little value in determining the nature of the undecomposed organic matter, except in the case of the sample of Hagerstown loam. This sample, taken from the "charcoal plot" of the Pennsylvania Experiment Station, was known to contain charcoal; and it will be noted in Table 5 that the quantity of undecomposed carbon is exceptionally large in this soil (13). The Ranger loam and the Lenox stony loam are peculiar soils in that they are derived from graphite schist. In samples of these soils the quantities of undecomposed carbonaceous matter are not large; hence it is improbable that graphite constitutes any considerable part of the carbon in the undecomposed residues of the other soil materials.

The preliminary work on the decomposition of various organic materials indicated that graphite, coal, and certain kinds of charcoal, if present in soils, would be found in the part not attacked by hydrogen peroxide. However, it is not to be expected that graphite, widely distributed in small quantities, would occur in anything more than traces in most soils. Coal particles would be restricted to coal-bearing regions. Because of the prevalence of forest fires, charcoal might be present in almost all soils. The decomposability of charcoals, however, is evidently variable.

Schreiner and Brown (11) found coallike and charcoallike particles in all of 34 soils examined by them, and also in samples from a number of deep subsoils. They point out that numerous forest fires furnish an ample source of charcoal, but add that it is not known how long the charcoal persists in the soil. White and Holben (14) showed that charcoal had persisted in the charcoal plot of the Pennsylvania experimental plots for more than 68 years. This particular plot was once the site of a charcoal furnace, and the soil on it now contains about 4 per cent of carbon. White and Holben found that charcoal in small quantities was a general constituent of all the Pennsylvania plots, and they consider charcoal a general, though small, constituent of soils in the eastern United States.

The residues from the hydrogen peroxide treatment were examined microscopically for evidence as to the nature of the undecomposed organic matter. Occasionally particles resembling roots were observed, but these particles seemed to be mere outlines and entirely different in appearance from roots not treated with hydrogen peroxide. In the Hagerstown loam, black particles, evidently charcoal, could be seen. The black particles gather on the sides of the beaker during the digestion. Charcoal can be distinguished by a test, devised by J. G. Smith of this laboratory, which consists of drawing a spatula firmly over a small quantity of soil material which has been placed on a clean, white sheet of paper. Charcoal gives a characteristic black streak. This may be confirmed by burning the black particles and by microscopic examination. White and Holben (14) recommend testing the soil for charcoal by stirring the soil in water; the charcoal particles will then rise to the surface.

Apparently there are forms of carbonaceous matter in the digested residues which do not possess the characteristics of charcoal, coal, or graphite. The residues from the peat and muck soil contained small quantities of a waxy substance which yielded dense, white fumes on burning. The odor and the fumes given off resembled those of paraffin. None of the other residues gave off white fumes on

burning. The fact that they blackened momentarily, indicated that a small quantity of organic matter was distributed through the residues. This momentary blackening may have been due to a trace of soluble organic matter which was not thoroughly removed by washing, since König, Hasenbäumer, and Grossmann (5) have found that acetic and formic acids are formed by the action of hydrogen peroxide on soil organic matter.

It is desirable to know the percentage of carbon in the carbonaceous matter of the undecomposed residues. It was found that further oxidation of the carbonaceous matter could be obtained by prolonged boiling of the residues in a closed flask with hydrogen peroxide. The flask was connected with a Knorr carbon dioxide apparatus and the carbon dioxide given off was weighed. Ten cubic centimeters of the 30 per cent peroxide was added and the contents of the flask were gently heated and finally boiled until no more carbon dioxide was given off, the process requiring several hours. Ten cubic centimeters more of peroxide was added, and this procedure was repeated until no more carbon dioxide could be obtained from the residues, 6 to 10 treatments being required. The boiled residue was then collected on a Gooch crucible, dried, and weighed, and the loss in weight noted. From the loss in weight of the sample and the weight of the carbon dioxide evolved the percentage of carbon in the carbonaceous matter destroyed was calculated. The carbon remaining after the prolonged boiling with hydrogen peroxide was determined by subtracting the quantity of carbon thus oxidized from the quantity of carbon present in the residue obtained in the standard method. These determinations, together with the carbon content of the residues from the first hydrogen peroxide treatment, are given in Table 6. Unless otherwise noted, the percentages are calculated on the weight of the oven-dried material rather than on the weight of the residue.

TABLE 6.—*Effect of prolonged boiling with hydrogen peroxide on the carbonaceous matter that remains after soils are treated by the standard peroxide method*

Type of soil or colloid	Quantity of carbon left after treatment by the standard peroxide method	Quantity of carbonaceous matter destroyed by prolonged boiling with hydrogen peroxide	Percentage of carbon in the carbonaceous material destroyed by prolonged boiling with hydrogen peroxide	Quantity of carbon left after prolonged boiling with hydrogen peroxide
	<i>Per cent</i>	<i>Per cent</i>		<i>Per cent</i>
Hagerstown loam.....	1.90	2.01	90	0.09
Carrington loam colloid.....	.41	.22	95	.20
Muck (surface layer).....	1.29	.52	73	.91

The quantities of carbon in the residues before and after prolonged boiling show that practically all the carbonaceous matter in the Hagerstown loam was decomposed by this treatment and that only from one-half to one-third of that contained in the Carrington loam colloid and muck was digested. The matter decomposed, being highly carbonaceous, is probably charcoal, while that unattacked is, presumably, not charcoal. These assumptions are supported by the fact that the carbon in the sample of Hagerstown loam, a soil material containing a large quantity of charcoal, was practically all

oxidized. Furthermore, a test showed that finely ground, nearly fresh charcoal was completely decomposed by prolonged boiling with peroxide.

It would seem from the above determinations that charcoal, or some material very high in carbon, usually constitutes a part of the carbonaceous matter that is left in the first treatment with hydrogen peroxide. Other more resistant carbonaceous substances may also be present. It is not known whether this resistant matter is high or low in carbon. The residues obtained in the analyses of Hagerstown loam, Houston black clay, and Sharkey clay by the standard method were found to contain insufficient nitrogen to give a test. It is probable, therefore, that the resistant matter contains no nitrogen.

It is not possible to state definitely the nature of the carbonaceous matter which resists decomposition by hydrogen peroxide in the adopted procedure. It is quite certain, however, that all organic matter of the nature of humus, crude fiber, and other cellulose material is decomposed. This is shown in the preliminary tests of these materials, and also by the fact that the residues from the peat and mosslike peat were very small. Preliminary tests with the adopted method indicate also a partial decomposition of charcoal or coallike material. From the microscopic examination it seems that a considerable part of the undecomposed organic matter in most of the materials examined may be charcoal, and graphite in two of the samples. In two cases a paraffinlike substance in the residues was indicated by ignition. Prolonged boiling with hydrogen peroxide also indicated that charcoal or a substance very high in carbon contained practically all the carbon of the undecomposed residue in one sample, and from one-half to one-third of the carbon in the residues of two other soils. Apparently the organic residues obtained from different soils contain different forms of carbon, and more than one kind of carbonaceous matter may be present in a single residue. In general, however, charcoal seems to be the most common carbonaceous constituent.

COMPARISON OF THE HYDROGEN PEROXIDE AND COMBUSTION METHODS OF DETERMINING ORGANIC MATTER

A comparison of the quantity of organic matter destroyed by the hydrogen peroxide method with the total organic matter determined by combustion is important. Although the former method is not accurate in determining the total organic matter in soils, since considerable carbon is found in the residues, the latter method is likewise inaccurate because of the uncertainty of the use of the factor for a particular soil. The hydrogen peroxide method has an advantage in that it gives a direct determination of the weight of the organic matter destroyed; and, since the quantity of undecomposed carbonaceous matter is small in many cases, it would seem that this method might give results as near the correct value as would the combustion methods.

Combustion determinations of organic matter were made on all the soils and colloids used in this investigation. The results of these combustion determinations are given in Table 7. In this table the organic matter determined by the hydrogen peroxide method is corrected for the carbonates present in the residues. The quantity of carbonaceous matter not decomposed by the peroxide is calculated

as carbon. The sum of the organic matter determined by hydrogen peroxide plus the quantity of carbon found in the residues is given for comparison with the combustion and hydrogen peroxide results. All percentages are calculated on the dry weight of the untreated material.

TABLE 7.—Comparison of organic matter of different soils as determined by the combustion method and the hydrogen peroxide method

Type of soil and colloid	Depth at which soil sample was taken	Quantity of organic matter determined by combustion (CO ₂ × 0.471)	Quantity of organic matter determined by hydrogen peroxide	Carbonaceous matter obtained from residue of peroxide digestion calculated as carbon	Organic matter obtained by hydrogen peroxide method plus the carbon in the residue	Organic matter as determined by combustion method compared with organic matter determined by hydrogen peroxide method plus the carbon in the residue
	Inches	Per cent	Per cent	Per cent	Per cent	Per cent
Amito loam.....	0-12	3.34	2.43	0.48	2.91	0.43
Do.....	12-36	2.30	1.70	.37	2.07	.23
Blakeley loam.....	0-12	2.91	2.43	.23	2.66	.25
Carrington loam.....	0-12	4.00	3.59	.30	3.89	.11
Carrington loam colloid.....	0-12	11.57	10.19	.41	10.60	.97
Cukre clay.....	0-5	9.56	9.05	.67	9.72	-.16
Do.....	5-60	2.49	2.25	.28	2.53	-.04
Hagerstown loam.....	0-8	9.22	4.92	1.90	6.82	2.40
Houston black clay.....	0-12	4.20	3.64	.60	4.24	-.04
Lenox stony loam.....	0-7	5.22	4.87	.50	5.37	-.15
Mosslike peat.....	0-6	85.60	95.57	.28	95.85	-10.25
Muck.....	0-6	24.47	23.16	1.29	24.45	.02
Do.....	6-12	14.09	12.61	.60	13.21	.88
Ontario loam.....	0-12	3.07	3.05	.06	3.11	-.04
Orangeburg fine sandy loam.....	10-36	.45	.36	.06	.42	.03
Orangeburg fine sandy loam colloid.....	0-10	4.28	3.63	.38	4.01	.27
Peat, from Florida.....	46-70	97.49	90.67	1.45	92.12	5.37
Palmar clay.....	0-14	7.38	6.87	.27	7.14	.24
Do.....	14-60	.87	1.06	.07	1.13	-.26
Do.....	60-72	.73	.85	.10	.95	-.22
Do.....	72-96	.68	.63	.06	.69	-.01
Do.....	96-120	.44	.51	.04	.55	-.11
Ranger loam.....	0-10	2.24	1.90	.32	2.22	.02
Sharkey clay.....	0-4	4.19	4.01	.44	4.45	-.26
Velasquez loam.....	12-32	15.06	13.17	.73	13.90	1.16
Wabash silt loam colloid.....	0-15	5.54	4.69	.50	5.19	.35

A comparison of columns 3 and 4 of Table 7 shows that the combustion method gives higher results than the hydrogen peroxide method in 22 of the 26 determinations, the differences being considerable in most cases. In 4 cases the hydrogen peroxide results exceed those obtained by the combustion method.

In some cases the ignition loss indicates which result is nearer the true value. In any case the ignition loss should exceed the quantity of organic matter, since it includes the combined water of the inorganic colloidal material and part of the sulphur and chlorine. The Florida peat has an ignition loss of 95.6 per cent. In this case the ignition loss must be nearly the correct value for organic matter, since very little water can be combined with inorganic material. Here the combustion value for organic matter, though obviously a little high, is more nearly correct than the hydrogen peroxide value. The ignition loss of the mosslike peat, 96.5 per cent, shows that, in

this case, the hydrogen peroxide value is the more accurate. Moreover, the fact that there is very little carbon in the undecomposed residues from this peat is another indication that the hydrogen peroxide value is very nearly correct. The muck subsoil has an ignition loss of 13.24 per cent, which is probably the correct value for organic matter, since the muck appears to be a mixture of quartz sand and organic matter, little or no water being combined with the organic material. The combustion value here is certainly too high, and the peroxide value is too low. In the case of the Hagerstown loam, the difference between the percentage of organic matter lost through ignition (9.52 per cent) and the percentage obtained by combustion (9.22 per cent) is only 0.30 per cent. This soil would contain ordinarily about 2.50 per cent of water combined with the inorganic colloidal material, since it contains 25 per cent of colloidal matter which averages 10 per cent of combined water. The ignition loss indicated in this case is 7.02 per cent of organic matter. The combustion value seems to be too high, therefore, by a difference of 2.20 per cent, and the peroxide value too low by a difference of 2.10 per cent. The ignition losses would seem to indicate that in one determination the combustion value is the more accurate, that in another determination the peroxide value is the more accurate, and that in two cases there is no choice between the two methods.

The correct value for organic matter is probably very close to the sum of the organic matter obtained by the hydrogen peroxide method, and the carbon in the residue; but it is probably slightly greater than this sum, since the carbonaceous matter in the residue is presumably not pure carbon. In any case this sum would represent the minimum quantity of organic matter present and would be only slightly less than the true value, as the quantity of carbon in the residue is small. The variation of the hydrogen peroxide values and the combustion values from this assumed correct value are shown in columns 5 and 7, respectively, of Table 7.

It is to be noted that in 11 cases there is no choice between the combustion and hydrogen peroxide methods, the values given by these methods differing by 0.16 or less from the assumed correct values. In 8 instances the combustion values are closer than the peroxide values, the average variation of the peroxide values and the maximum variation being 0.55 per cent, and 1.29 per cent, respectively, of the weight of the soil. In 7 determinations the peroxide results are nearer the assumed correct values. The combustion values in these 7 cases differ by an average of 3.04 per cent, and show a maximum deviation of 10.25 per cent.

It is apparent that the peroxide method gives about as accurate results, although usually low, as the combustion method. In the case of somewhat unusual soil materials also the results may be considerably more accurate.

ACCURACY WITH WHICH CONVENTIONAL FACTOR OF 0.471 APPLIES TO ORGANIC MATTER DESTROYED BY HYDROGEN PEROXIDE

It has just been shown that some of the combustion values for organic matter are more or less incorrect. Obviously the reason for this is that the factor used for calculating the carbon dioxide to organic matter does not correctly represent the composition of the organic matter of certain soils.

The use of the factor, 0.471, was first proposed by Wolff (15), in 1864. He found that there was, on the average, 58 per cent of carbon in the humus extracted from soils and in artificial humus preparations. To get the weight of organic matter, therefore, he recommended multiplying the quantity of carbon dioxide evolved on oxidation by the factor, 0.471. This factor has been used for the determination of organic matter in soils ever since.

It is clear that a factor derived from the average composition of soil organic matter may be quite inaccurate when applied to any particular soil, although it may correctly represent the average composition of the organic matter of a number of soils. From a large number of subsequent determinations, however, it is apparent that there may be considerably less than 58 per cent of carbon in the average soil organic matter. For instance, the average percentage of carbon in the humus extract of 40 soils, as given by Eggertz (3), Cameron and Breazeale (1), and Fraps and Hamner (4) is 45.6. This evidence is not conclusive, since the humus extract is only a part of the organic matter, and its composition may not be representative of all the organic matter present. Snyder (12) and Read and Ridgell (8) determined the carbon in the entire organic matter in 81 samples of soil. The average percentage of carbon in these 81 samples was 49.4.

The above averages indicate that the factor, 0.471, may be too low for the average soil. Further evidence regarding the accuracy of this factor is evidently desirable. The data in Table 7 bear on this point. By the use of these data, it is possible to calculate the percentage of carbon in most of the organic matter in each of the 26 samples examined. The hydrogen peroxide destroyed almost all the organic matter in many of these soil materials. The weight of the organic matter destroyed in each case is known, and the percentage of carbon in the organic matter may be calculated by subtracting the carbon in the residue from the total carbon, as determined by combustion, and dividing the remainder by the organic matter decomposed by hydrogen peroxide. The proportions of carbon calculated in this way are given in column 7 of Table 8.

The figures given in Table 8 show that the percentage of carbon in the organic matter decomposed by hydrogen peroxide varies considerably, ranging from 37.5 to 70.1, with an average of 54.8 per cent. Of the 26 samples tested, 5 samples (Nos. 15, 19, 20, 21, and 22) show determinations of slight dependability on account of the low organic content; and one soil, the Hagerstown loam, gives an abnormally high carbon percentage, on account of the artificial addition of charcoal. The remaining 20 samples cover a wide range of soils, including as they do, 2 peats, 2 mucks, and 3 soil colloids. Excluding Nos. 8, 15, 19, 20, 21, and 22, for the reasons noted above, the average carbon content for the remaining 20 is 56.3 per cent, with a minimum of 49.8 per cent and a maximum of 61.9 per cent.

When all the results of digestion by hydrogen peroxide are considered, the average percentage of carbon is considerably higher than the averages reported by investigators subsequent to Wolff. In the selected list, the average is still higher, being fairly close to the 58 per cent required to make Wolff's (15) conventional factor applicable.

TABLE 8.—Quantity of carbon in the organic matter of various types of soil destroyed by hydrogen peroxide

Sample No.	Type of soil and colloid	Depth at which soil sample was taken	Quantity of organic matter as determined by decomposition with hydrogen peroxide	Quantity of combustible matter calculated as carbon in residue from H ₂ O ₂ treatment	Quantity of carbon in total organic matter of soil ¹	Quantity of carbon in organic matter destroyed by hydrogen peroxide
		Inches	Per cent	Per cent	Per cent	Per cent
1	Amito loam.....	0-12	2.43	0.48	66.5	59.9
2	do.....	12-36	1.70	.37	64.4	57.0
3	Blakeley loam.....	0-12	2.43	.23	63.2	59.9
4	Carrington loam.....	0-12	3.59	.30	59.6	56.2
5	Carrington loam colloid.....	0-12	10.19	.41	63.3	61.9
6	Cukre clay.....	0-5	9.05	.67	57.0	53.9
7	do.....	5-60	2.25	.28	59.4	54.1
8	Hagerstown loam.....	0	4.92	1.90	78.4	70.1
9	Houston black clay.....	0-12	3.64	.60	57.5	50.5
10	Lenox stony loam.....	0-7	4.87	.50	55.3	50.6
11	Mosslike peat.....	0	95.57	.28	51.8	51.9
12	Muck.....	0-6	23.16	1.29	58.0	55.7
13	do.....	6-12	12.61	.60	61.8	60.1
14	Ontario loam.....	6-12	3.05	.06	57.2	56.5
15	Orangeburg fine sandy loam.....	10-36	.36	.06	62.1	56.4
16	Orangeburg fine sandy loam colloid.....	0-10	3.63	.38	61.9	58.0
17	Peat.....	46-70	90.67	1.45	61.3	60.8
18	Palmar clay.....	0-14	6.87	.27	59.8	58.3
19	Do.....	14-60	1.06	.07	44.6	41.0
20	Do.....	60-72	.85	.10	44.5	37.5
21	Do.....	72-96	.63	.06	57.4	53.4
22	Do.....	96-120	.51	.04	46.4	42.1
23	Ranger loam.....	0-10	1.90	.32	58.5	51.3
24	Sharkey clay.....	0-4	4.01	.44	54.6	49.8
25	Velasquez loam.....	12-32	13.17	.73	62.1	60.8
26	Wabash silt loam colloid.....	0-15	4.69	.50	61.9	57.9
Average, all samples.....					58.6	54.8
Average, excluding Nos. 8, 15, 19, 20, 21, and 22.....					59.7	56.3

¹ It is assumed that the organic matter not decomposed by H₂O₂ is carbon.

Although the preceding calculation applies to a larger part of the total organic matter than the calculations of Wolff, Eggertz, and others who have worked on humus extracts, it does not apply completely to the total organic matter, since a small quantity is not decomposed by hydrogen peroxide. There is some evidence that the organic matter not decomposed by hydrogen peroxide is higher in carbon than that which is decomposed. If this is so, it would raise the percentage of carbon in the total organic matter, in each case to a figure slightly higher than that expressed by the figure in column 6, Table 8. On the assumption that the organic matter in the residues is all carbon, the percentage of carbon in the entire organic matter of the soil was calculated. The results are given in column 7 of Table 8. The average carbon content for the 20 selected soils is 59.7 per cent. Doubtless the true average percentage for the total organic matter lies at some point between 56.3 and 59.7 per cent of carbon. It can thus be seen that the conventional factor, 0.471, corresponding to 58 per cent carbon, applies very well to the average organic matter in the soil types used in this study.

In individual cases the application of the factor leads to comparatively large errors, the maximum being 10 per cent. However, these errors occurred in soils so very high in organic matter that the combustion method is not ordinarily applied to them. The Hagerstown loam is an exception, because the large quantity of charcoal present in it is responsible for considerable error. From these results, it

seems reasonable to conclude that occasionally large errors may result from the use of the conventional factor. In general, however, this factor seems to represent the composition of organic matter rather accurately.

LIMITATIONS AND ADVANTAGES OF THE HYDROGEN PEROXIDE METHOD

From the point of view of manipulation the hydrogen peroxide method is satisfactory. The agreement of duplicates is good. The basic idea is simple, though fully as much care must be given to details of procedure as in the combustion method. The hydrogen peroxide method offers no advantages so far as rapidity of determination is concerned, over the combustion method, except when a large number of analyses are to be made.

The hydrogen peroxide method is not applicable to soils which contain more than 2 or 3 per cent of calcium carbonate or more than a trace of manganese dioxide or chromium sesquioxide. Soils containing chromium sesquioxide are rare; and soils containing more than a trace of manganese dioxide are, as a rule, confined to the tropical or subtropical latitudes.

As a means of determining the total organic matter in soils, the hydrogen peroxide method shows little, if any, inferiority to the combustion method. The peroxide method always gives low results; but the errors are small in most cases. The combustion method, on the other hand, sometimes gives too high or too low results, and occasionally the errors are large.

Although hydrogen peroxide does not completely decompose the organic matter in the soil, there is no good evidence that it determines any clearly defined kind of organic matter. The so-called "humus" is entirely destroyed; but since peat, crude fiber, and other cellulose materials are likewise decomposed by hydrogen peroxide, the method obviously can not be taken as a means for determining the humus in soils. Neither can the part of the soil which is not decomposed by peroxide be regarded as containing all the charcoal originally present, for under some conditions considerable charcoal is decomposed.

It is presumed that the carbonaceous matter remaining in the residues from the peroxide treatment is inert and hard to oxidize by biological processes. Probably this unattacked carbonaceous matter is not so important in soil fertility as the organic matter which is attacked by hydrogen peroxide. On the other hand, the peroxide method obviously can not be used to determine the readily available organic matter.

For routine analysis, the hydrogen peroxide method offers no advantage over the combustion method. However, in investigations where an accurate determination of the organic matter is necessary, as in the determination of the water which is combined with the inorganic material of soils, a combination of the hydrogen peroxide and combustion methods offers distinct advantages. If the carbon in the residue from the hydrogen peroxide digestion were determined by combustion and then added to the organic matter determined by hydrogen peroxide, the sum would be a more accurate figure for the total organic matter than the value obtained by any of the present methods.

SUMMARY

A report is given of the possibility of determining the organic matter in soils by the loss in weight caused by digestion with hydrogen peroxide, and a method for this determination is described.

Several forms of organic matter, including cellulose, humus, and lignite, are almost completely decomposed by hydrogen peroxide in the presence of soil. Graphite is unattacked and charcoal and coal are partially decomposed.

Determinations made by the hydrogen peroxide method on a variety of soils and colloids containing from 0.42 to 95.85 per cent of organic matter show that practically all the organic matter is decomposed in some soils, and that in other soils considerable organic matter is unattacked.

The nature of the carbonaceous matter resisting decomposition by hydrogen peroxide is uncertain. In some soils, charcoal forms a major part of the undecomposed organic matter, but other organic matter may also be present.

Although the hydrogen peroxide method gives low results, it is, on the whole, about as accurate as the combustion method, and may be considerably more accurate in special cases.

The method gives some indication concerning the percentage of carbon in soil organic matter. In the 26 samples analyzed, the organic matter averaged about 58 per cent carbon. This is the proportion of carbon required by the conventional factor, 0.471, used for calculating the carbon dioxide evolved by combustion to organic matter. Individual cases show considerable variation from this average of 58 per cent.

The hydrogen peroxide method is not applicable to soils high in calcium carbonate, manganese dioxide, or chromium sesquioxide.

Apparently the hydrogen peroxide method does not determine any clearly defined kind of soil organic matter. It decomposes more than the so-called "humus," and is therefore not a method to be used in determining the humified organic matter in the soil.

For routine analysis, the hydrogen peroxide method offers no advantages over the combustion methods. It should, however, prove useful in investigations, since this method, when it is supplemented by a combustion determination of carbon in undecomposed residues, should give the most accurate value for soil organic matter.

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